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(19)

(54) A METHOD OF PREPARING GRANULATED VITAMINIZED COATED MINERAL FOOD COMPLEMENTS FOR ANIMALS, AND PRODUCTS THUS OBTAINED

(71) We, S.A.R.A.P. "CEDIA", of rue du Petit Bièvre, 91-Bievres, France, a French Body Corporate, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a method of producing improved granulated vitaminized coated mineral food complement for animals.

For technical and economic reasons, the demand from stock breeders for animal food complementary products is steadily increasing, especially for vitaminized mineral premixes. The main advantage of these complementary or supplementary products is that they make use of local farming resources, such as cereals, maize, beets and pulp, thus correspondingly reducing the production expense.

For many years the mineral mixtures were provided in powder form. However, as is usual in the case of powdered products, the animals' appetite for these products was very poor and in many cases the animals refused to eat them. Because of this problem it has been suggested that beetroots, Jerusalem artichokes or bran should be incorporated in the basic foodstuff. This method of dispensing the food, however, involves the cost of additional labour and was only applicable in the cattle shed and in winter.

During the last few years, various attempts have been made to solve the problem by granulating the vitaminized mineral premix. Granulation has been achieved in conventional presses, frequently by using specially adapted dies. To obtain satisfactory results the following requirements had to be met:

1) the granules should have a relatively small diameter;
2) the granules should be prevented from becoming too hard; and
3) the product must be palatable.

The following disadvantages were observed:

a) high pressure granulation was accompanied by a temperature rise of 45 to 50°C, under which conditions vitamins A and D were usually destroyed;
b) a vegetable support or carrier representing from 20 to 25% of the formulation had to be incorporated in the product, thus reducing the proportion of total mineral substances in the product; and
c) the presence of this vegetable matter not only caused problems of fermentation and mould, but limited the product to use under shelter or in the stable.

Finally, both the production of powdered products and the production of granules as described above entailed the use of natural or synthetic aromatic or flavouring substances to improve the palatability and to conceal the odour of certain constituents.

There thus remained a need for a process for preparing a feed complement which:

1) could produce granules of varied composition for the various types of animal to be fed, without the use of a press;
2) improve the palatability of the product to animals without adding the conventional flavouring substances, thus reducing cost;
3) increase the content of minerals and hence the intrinsic value of the produce; and
4) provided compounds which could be dispensed both in the open-air and in the cattle shed.

The present invention is based on the use of mineral products in a particulate form, such as granules, in particular phosphates in the form of monocalcium and/or dicalcium and/or mixed particulate mono- and dicalcium phosphates which have been treated to

cause them to expand and take the form of porous particles with a diameter of 0.2 to 2.0 mm and which are capable of absorbing considerable amounts of liquid, especially from 8 to 10% of their weight. This capability of absorbing liquids is used as the basis of the present invention.

According to the present invention we provide a method of preparing palatable granulated vitaminized coated mineral feed complements, in which monocalcium phosphate and/or dicalcium phosphates, in the form of porous particles or granules having a particle size within the range of 0.2 to 2 mm capable of absorbing a liquid is introduced into a slow speed mixer at room temperature, wherein during a first step, trace elements and vitamins in powder form are added; then, after mixing for a few minutes, in a second step an appetite promoting liquid selected from molasses, glucose syrups, yeast autolysates, soya-bean lecithin, and mixtures thereof, is added; and finally, after a further few minutes' mixing in a third step a desiccating coating substance is added, said third step lasting 1 to 2 minutes; the granules thus obtained containing said trace elements and said vitamin powder encrusted in their porous surface, whereas said appetite promoting liquid is absorbed in the pores of said surface, with the aforesaid desiccating substance coating the said granules.

The method is conveniently effected using a conventional mixer, or preferably, a horizontal slow-rotating kneading machine. The mixer is supplied with calcium phosphate granules and then with trace elements and powdered vitamins in the form of a concentrate mixture in a carrier, while limiting as far as possible the amount of said carrier. After the ingredients have been mixed for about 2 minutes, the trace elements and vitamins are encrusted in the cells of the porous walls of the phosphate granules, and substantially no free powder is left in the machine. Then, the selected appetite-promoting liquid is introduced by using either an injector or a range of nozzles, or a simple, relatively fine, jet. The liquid is absorbed after mixing for 2 or 3 minutes, the machine being operated during this working step. Thus, a homogeneous distribution is obtained and the final step of the operation consisting in drying the mixture by absorbing the residual moisture on the particle surface is carried out. To this end, from 2 to 12% (according to the amount of liquid incorporated) of the desiccating coating substance, preferably anhydrous dicalcium phosphate or anhydrous magnesium hydroxide, are used. The working time, for this last step, is relatively short, i.e. 1 or 2 minutes.

The fixation and penetration of the liquid may be improved by adding to the liquid 0.5% of liquid sorbitol. The function of sorbitol is to soften the molasses or lecithin while improving the penetrating capacity.

Of course, the use of mono- and dicalcium phosphates in the form of expanded granules will not provide in all cases the desired P/Ca ratios. However, it is possible to correct the mixture compositions by adding calcium in the form of a granulated salt or marine limestone; if desired, magnesium in the form of a salt or granulated oxide may also be added. Furthermore, particulate ammonium phosphate may also be added in the form of mono-, di- or triammonium phosphate, or mixtures thereof, as well as sodium chloride. In the manufacturing process these substances are added before introducing the liquid intended to improve the animals' appetite into the mixer.

Now three typical formulae corresponding to P/Ca weight ratios as specified below, and based on the use of expanded monocalcium phosphate will be given, the numerals designating the percentages by weight in relation to the total of components:

Biological equilibrium	P/Ca 20/15	P/Ca 19/12	P/Ca 14/14
Total mineral substance %	70	70	65
Minimum P	20	19	14
Ca	15	12	14
Maximum NaCl	3	2	2
Insoluble residue	2	2	3

The following two formulae are based on the use of expanded dicalcium phosphate:

Biological equilibrium	P/Ca 14/14	P/Ca 12/18
Total mineral substances %	62	65
Minimum P	14	12
Ca	14	18
Maximum NaCl	4	2
Insoluble residue	3	2.5

This appetite-promoting coating method, carried out by using molasses, glucose syrups, yeast autolysates or lecithin, can yield active products of the veterinary type.

A few examples of vitaminized mineral compositions for animals, in the form of granules according to this invention will now be given. The parts and percentages are given by weight. The numerals associated with the vitamins designate the number of international units (IU) of vitamins per gram of the vitamin substance utilized and the numerals associated with the mineral salts and oxides designate the percentage by weight of the cation in the mineral salt utilized. The compositions may be prepared by the process described above.

Example 1

Mineral compound with vitamins A, D_3 and E, with the addition of molasses.

(P/Ca=17/17) for bovine, ovine and caprine cattle:

Granulated monocalcium phosphate	73.000
Trace elements	0.500
Vitamins A 100,000	0.220
Vitamins D_3 100,000	0.055
Vitamins E 25	0.010
Pulverulent calcium carbonate	0.215
Granulated magnesium sulfate 32	5.000
Granulated calcium carbonate	7.000
Molasses	8.000
50% star anise	0.500
Anhydrous dicalcium phosphate	6.000
Iron oxide	0.500
	101.000

Guarantee per 100 kg

Minimum:	
Total mineral substances	65%
Phosphorus	17%
Calcium	17%
Maximum:	
NaCl	2%
Matter insoluble in hydrochloric acid	2%

Vitamins per 100 kg

A	20,000,000
D_3	5,000,000
E	2,000 mg

Example 2

Vitaminized mineral compound with vitamins A and D_3, and the addition of molasses and yeast autolysates.

(P/Ca: 20/15) for bovine, ovine and caprine cattle:

Granulated mooncalcium phosphate	84.000
Magnesium sulfate (granular) 32	2.000
Trace elements	0.500
Vitamin A 100,000	0.200
Vitamin D_3 100,000	0.050
Vitamin E 25	0.008
Molasses	6.000
Yeast autolysates	2.000
Sorbitol	0.500
Anhydrous dicalcium phosphate	5.000
Iron monoxide	0.600
50% star anise	0.500
	100.958

Guarantee per 100 kg

Minimum:	
Total mineral substances	70%
Phosphorus	20%
Calcium	15%
Maximum:	
NaCl	2%
Hydrochloric insoluble matter	2%

Vitamins per 100 kg

A	20,000,000
D_3	5,000,000
E	2,000 mg

Example 3

Mineral compound with vitamins A & D_3 and the addition of molasses and lecithin.

(P/Ca 14/14) for bovine, ovine and caprine cattle:

Granulated monocalcium phosphate	55.000
Trace elements	0.500
Vitamins A 100,000	0.100
Vitamins D_3 100,000	0.080
Magnesium sulfate (granular) 32	10.000
Calcium carbonate (granular)	10.000
Molasses	10.000
Lecithin	2.000
Anhydrous dicalcium phosphate	12.000
Iron monoxide	0.500
Star anise	0.250
	100.430

Guarantee per 100 kg

Minimum:	
Total mineral matters	70%
Phosphorus	14%
Calcium	14%

Maximum:	
NaCl	2%
Matter insoluble in hydrochloric acid	2%
Vitamins per 100 Kg	
A	10,000,000
D_3	8,000,000

Example 4

Mineral compound with vitamins A, D_3 and E for bovine, ovine and caprine cattle, with the addition of molasses.

Bicalcium phosphate granules	73.000
Trace elements	0.500
Vitamins A 100,000	0.220
Vitamins D_3 100,000	0.050
Vitamins E 25	0.010
Magnesium sulfate (granular) 32	5.000
Molasses	15.000
50% star anise	0.500
Anhydrous dicalcium phosphate	6.000
	100.280

Guarantee per 100 kg	
Minimum	
Total mineral matters	63%
Phosphorus	15%
Calcium	16%
Maximum	
Chlorides (NaCl)	2%
Matter insoluble in hydrochloric acid	2%
Vitamins per 100 kg	
A	20,000,000
D_3	5,000,000
E	2,000 mg

Example 5

Nitrogeneous mineral compound in the form of molasses-containing granules.

Granulated monocalcium phosphate	13%
Granulated monoammonium phosphate	32%
Granulated magnesium oxide	20%
Chloride (NaCl)	3%
Granulated calcium carbonate	25%
Trace elements	0.5%
Vitamin pre-mix	0.5%
Molasses	4%
Anhydrous dicalcium phosphate	2%
	100%

Guarantee per 100 kg	
Minimum	
Total mineral matters	85%
Phosphorus (P)	10%
Calcium (Ca)	10%
Maximum	
NaCl	5%
Matter insoluble in hydrochloric acid	3%
Equivalent of protein substances	20%

The following is a description of other additives that can be substituted for those mentioned in the preceding Examples:

I. — A suitable additive consists of a glucose syrup constituting an adequate substitute for molasses, and assaying as follows by weight:

Dextrose	64%
Maltose	13%
Isomaltose	4%
Triose	6%
Tetraose	5%
Superior sugar	8%

The essential feature characterising this additive is that it is available in both dry and liquid forms. It is therefore much easier to control the total moisture content than in the case of molasses. On the other hand, this product has a considerably better and more pleasant sweetening or sugaring capacity, thus improving appreciably the animals' appetite for mineral food complements.

Finally, due to its viscosity, this additive reduces considerably the percentage of fines or powder, thus affording an increment in the granule diameter or size by either extending the mixing time or alternating the addition of glucose syrup and anhydrous dicalcium phosphate.

II. — In the above formulae for mineral compounds the complementary calcium was in the form of powdered or granular calcium carbonate.

Now, there is a well-known natural source of calcium, namely the marine calcareous or limestone deposits in the form of chalks or small madrepores. These are known for instance in Brittany under the name of "Maerl". This product can be crushed, ground, calibrated and dehydrated and becomes particularly absorbent and porous, so that homogeneous mixtures can be prepared therefrom with the other constituents. The above-defined "Maerl" substance contains traces of natural trace elements and these obviously improve the value of the products.

A few examples concerning formulae of Maerl-containing food complements are given hereinafter, the numerals designating the percentages by weight.

Example 6

Maerl-containing compound

Granulated monocalcium phosphate	25
Granulated dehydrated Maerl	40
Salt (NaCl)	14
Iron sulfate 21	1
Zinc oxide 64	0.44
Manganese oxide 62	0.250
Cobalt sulfate 21	0.010
Stabilized potassium iodide	0.002
Granulated magnesium oxide 85	7
Molasses	7
Anhydrous dicalcium phosphate	5
+Vitamins A, D_3, E, B, K...	0.300
	100.002

Guarantee per 100 kg

Minimum

Total mineral substances	80%
Phosphorus	6.5%
Calcium	15%

Maximum

Chloride (NaCl)	17%
Matter insoluble in hydrochloric acid	4%

Example 7

"Maerl" formula compound

Granulated monocalcium phosphate	41.000
Granulated dehydrated Maerl	27.000
Salt (NaCl)	1.500
Zinc sulfate 33	1.500
Iron sulfate 21	1.000
Copper sulfate 25	0.500
Manganese sulfate 28	0.500
Cobalt sulfate 21	0.010
Stabilized potassium iodide	0.002
Anhydrous sodium sulfate	2.000
Granulated magnesium sulfate 32	15.000
Molasses	5.000
Anhydrous dicalcium phosphate	5.000
	100.000

Guarantee for 100 kg

Minimum

Mineral substances	85%
Phosphorus	10%
Calcium	16%

Maximum

Chloride (NaCl)	3%
Matter insoluble in hydrochloric acid	2%

III. — For dietetic, veterinary or paraveterinary products such as "Diafer" (containing Fe^{++}), a suitable formula containing ferrous iron introduced at the same time as the trace elements is given hereinafter, the percentages being by weight:

Example 8

Granulated monocalcium phosphate	50.000
Glucose syrup	7.000
Yeast autolysates	10.000
Iron fumarate (Fe^{++})	2.750
Copper fumarate	0.250
Vitamin A 100,000	0.200
Vitamin D_3 100,000	0.020
Vitamin B_{12} (1000 mg/kg)	0.100
Sodium propionate	0.200
Synthetic aroma	0.200
Anhydrous dicalcium phosphate	30.000
	100.720

The Fe^{++} contents may vary to a larger extent as a function of the carrier (from 2.700 g to 80 g or more).

It should be understood that the chemical and physical properties (such as granulometry and absorbent capacity) of monocalcium phosphates and dicalcium phosphates in the particulate form, coated with a sugaring substance, can be used for preparing certain veterinarian products, for example with a view to improve the palatability of certain veterinary substances to animals. Therefore, the same monocalcium and dicalcium phosphate in the expanded state, coated with sugaring substances, may be used for developing products and substances such as dietetics, vermifuges, antianemic, antidiarrhoea, antistress, metabolism-regulating and antiinfectious substances.

IV. — Fatty substances.

Recent work proved that there is a possibility of causing particulate monocalcium phosphate to absorb fatty substances of miscellaneous origins in the form of oil or solutions.

This property permits notably:

1) of fixing on the phosphate fatty substances of which the presence is subsequently required in the compound food (notably for poultry);

2) of utilizing veterinary products in an oily form to facilitate their use and commercialization;

The technique contemplated for incorporating such fatty substances is that already described in connection with the absorption of a liquid; the finished product is dried and protected against lumping by using anhydrous dicalcium phosphate and possibly a calcium silicate.

The rate of fatty substances fixed by the monocalcium phosphate may vary from about 1 to 10% as a function of the specific nature of the fatty substances used.

It may also be pointed out that instead of the monocalcium and dicalcium phosphates in the form of porous particles used in the mineral compounds of this invention, any other calcium phosphate in the form of absorbent porous particles may be used, notably mixed monocalcium and dicalcium phosphates in the form of porous particles.

In fact, these mixed calcium phosphates having undergone a suitable treatment and presented in the form of porous granules having an absorbent power are commercially available.

These mixed calcium phosphates can be produced by varying the purified phosphoric acid percentage, notably by reducing the acid-to-calcium ratio or proportion, when attacking the calcium with said phosphoric acid. Thus, for example, mixed calcium phosphates containing 60 to 70% of monocalcium phosphate and 40 to 30% of dicalcium phosphate may be produced. These phosphates may assay, for instance, 20 to 21% phosphorus and 20 to 21% calcium. They constitute stable products having an appearance similar to that of pure particulate monocalcium phosphate or pure particulate dicalcium phosphate, and have similar properties. Therefore, they are perfectly suited for preparing food complements of the type set forth hereinabove and may be used for preparing these compounds either alone or in admixture with particulate monocalcium phosphate and/or particulate dicalcium phosphate.

Two formulae of food complements containing mixed phosphates are given hereinafter by way of example. These compositions should not be construed as limiting the present invention, since the calcium phosphate percentages may be varied as well as that of other components, and it is also possible to add calcium and/or magnesium in the form of salts or oxides for modifying the phosphorus, calcium and magnesium contents of the food complements thus prepared.

Example 9

Food complement containing 16.5% phosphorus and 17.2% calcium.

Mixed monocalcium and dicalcium phosphate in particulate form (20% phosphorus and 20% calcium)	80.000
Trace elements and vitamins	1.500
Particulate magnesium oxide	7.000
Kitchen salt (NaCl)	3.000
Molasses or sugar	4.500
Anhydrous dicalcium phosphate	4.000
	100.000

Example 10

Food complement containing 17% phosphorus and 13.5% calcium.

Mixed monocalcium and dicalcium phosphate in granular form (with 20% phosphorus and 20% calcium)	30.000
Particulate monocalcium phosphate	50.000
Trace elements and vitamins	1.500
Particulate magnesium oxide	5.000
Kitchen salt (NaCl)	5.000
Molasses	4.500
Anhydrous dicalcium phosphate	4.000
	100.000

WHAT WE CLAIM IS:—

1. A method of preparing palatable granulated vitaminized coated mineral feed complements, in which monocalcium phosphate and/or dicalcium phosphate and/or mixed monocalcium and dicalcium phosphates, in the form of porous particles or granules having a particle size within the range of 0.2 to 2 mm capable of absorbing a liquid is introduced into a slow speed mixer at room temperature, wherein during a first step, trace elements and vitamins in powder form are added; then, after mixing for a few minutes, in a second step an appetite promoting liquid selected from molasses, glucose syrups, yeast autolysates, soya-bean lecithin, and mixtures thereof, is added; and finally, after a further few minutes' mixing in a third step a desiccating coating substance is added, said third step lasting 1 to 2 minutes; the granules thus obtained containing said trace elements and said vitamin powder encrusted in their porous surface, whereas said appetite promoting liquid is absorbed in the pores of said surface, with the aforesaid desiccating substance coating the said granules.

2. A method according to Claim 1, in which there are introduced into the mixer between said first step and said second step, various additives selected from granulated calcium salts, particulate marine limestone, particulate magnesium salts and oxides, sodium salts, particulate ammonium phosphate in the form of monoammonium phosphate or diammonium phosphate or tri-ammonium phosphate, or mixtures of these forms, and mixtures of these substances; these additives being mixed with the particulate calcium phosphate containing said trace elements and vitamins, before introducing said appetite promoting liquid into the mixer in the second step.

3. A method according to any of Claims 1 or 2, in which liquid sorbitol is introduced in admixture with said appetite promoting liquid.

4. A method according to Claim 3, in which 0.5% by weight of liquid sorbitol in

relation to the weight of the final granulated product is introduced.

5. A method according to any of Claims 1 to 4, in which 2 to 12% by weight of said desiccating substances in relation to the weight of the final granulated product are introduced.

6. A method according to any of Claims 1 to 5, in which the desiccating coating substance is anhydrous dicalcium phosphate or anhydrous magnesium hydroxide.

7. A method according to Claim 2, in which 73% of particulate monocalcium phosphate, 5% of particulate magnesium sulfate, 7% of particulate calcium carbonate, 8% of molasses, and 6% of anhydrous dicalcium phosphate are used, these percentages relating to the weight of final granulated product.

8. A method according to Claim 2, in which 84% by weight of particulate monocalcium phosphate, 2% of particulate magnesia sulfate, 6% of molasses, 2% of yeast autolysates, 0.5% of sorbitol, and 5% of anhydrous dicalcium phosphate are used, these percentages relating to the weight of the final granulated product.

9. A method according to Claim 2, in which 55% of particulate monocalcium phosphate, 10% of particulate magnesium sulfate, 10% of particulate calcium carbonate, 10% of molasses, 2% of lecithin, and 12% of anhydrous dicalcium phosphate are used, these percentages referring to the weight of the final granulated product.

10. A method according to Claim 2, in which 73% of particulate dicalcium phosphate, 5% of particulate magnesium sulfate, 15% of molasses, and 6% of anhydrous dicalcium phosphate are used, these percentages referring to the weight of the final granulated product.

11. A method according to Claim 2, in which 13% of particulate monocalcium phosphate, 32% of particulate monoammonium phosphate, 20% of particulate magnesium oxide, 3% of sodium chloride, 25% of particulate calcium carbonate, 4% molasses and 2% anhydrous dicalcium phosphate are used, these percentages referring to the weight of final granulated product.

12. A method according to Claim 2, in which 25% of particulate monocalcium phosphate, 40% of particulate marine limestone, 14% of sodium chloride, 7% of particulate magnesium oxide, 7% of molasses, and 5% of anhydrous dicalcium phosphate are used, these percentages referring to the weight of the final granulated product.

13. A method according to Claim 2, in which 41% of particulate monocalcium phosphate, 27% of particulate marine limestone, 1.5% of sodium chloride, 15% of particulate magnesium sulfate, 2% of sodium sulfate, 5% f molasses, and 5% of anhydrous dicalcium phosphate, are used, these percentages referring to the weight of final granulated product.

14. A method according to Claim 1, in which 50% of particulate monocalcium phosphate, 7% of liquid glucose syrups, 10% of yeast autolysates, and 30% of anhydrous dicalcium phosphate are used, the percentages referring to the weight of the final granulated product.

15. A method according to Claim 2, in which 80% of particulate calcium phosphate consisting of mixed monocalcium and dicalcium, (with 20% phosphorus and 20% calcium), 7% particulate magnesium oxide, 3% sodium chloride, 4.5% molasses or sugar, and 4% anhydrous dicalcium phosphate are used, the percentages referring to the weight of the final granulated product.

16. A method according to Claim 2, in which 30% of particulate calcium phosphate consisting of mixed monocalcium and dicalcium (with 20% phosphorus and 20% calcium), 50% of particulate monocalcium phosphate, 5% particulate magnesium oxide, 5% of particulate sodium chloride, 4.5% of molasses, and 4% of anhydrous dicalcium phosphate are used, the percentages referring to the weight of the final granulated product.

17. A method according to any of Claims 1 to 16, in which the porous particles or granules of calcium phosphates used are capable of absorbing from 8 to 10% of the weight of the liquid.

18. Granulated vitaminized coated mineral feed complements for animals, obtained by carrying out a method as claimed in any of Claims 1 to 16.

19. A method of preparing a granulated, coated feed supplement according to claim 1 using porous particles or granules of calcium phosphates, substantially as herein described.

20. A modification of the method according to claim 1 in which the appetite promoting liquid is replaced by a fatty substance.

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